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**Boron geochemistry of the Miocene-Quaternary  
calc-alkaline volcanic rocks of the  
Carpathian Pannonian Region and its relation to  
subduction processes:  
prompt gamma activation analytical measurements**

SUMMARY OF THE DOCTORAL (PhD) DISSERTATION

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## I. INTRODUCTION, AIMS

Most volcanic structures (90%) are built on the border of the solid outer layer of the Earth. The magmatic processes of the convergent plates are influenced by the type of subducted sediments, the age, the temperature of the slab, the fluids released from it and also by the geochemical and petrological features of the mantle wedge. Volcanic rocks of the continental margins are richer in fluid mobile and incompatible elements than the mantle rocks. Subduction zones are the best laboratories to study the chemical connection and transfer of materials between the crust and the mantle. Subduction zone related volcanic rocks contain some trace elements and trace element ratios, which can sensitively reflect the deep chemical processes.

In recent decades more and more researchers study the distribution of the fluid mobile elements like boron to learn the material recycling of the subduction zones and volcanism. Boron is not only fluid mobile, but is also an incompatible element, and thus is a good tracer of fluid metasomatism (e.g. Morris et al. 1990; Ryan & Langmuir 1993). However, the geochemical importance of boron was realised only in the beginning of the 1990's, with the advent of the systematic boron concentration measurements of volcanic and metamorphic rocks. The reason for the late date was the difficulty in the precise measurement of the boron content in whole rocks. Nowadays more than one method is used to measure the boron content of bulk rocks, including inductively coupled plasma- (ICP-MS), and secondary ion mass spectrometry (SIMS). The most suitable and precise analytical technique to measure B concentration without sample preparation is prompt gamma activation analysis (PGAA).

In Europe there is only one PGAA instrument (Budapest, Hungary) which has performed boron and other trace and major element analysis on geological samples, which were then published in international scientific journals. The PGAA instrument is operated by the Nuclear Research Department of the Institute of Isotopes HAS. As a colleague of the department, I measured for the first time the calc-alkaline volcanic samples from the Carpathian Pannonian Region with PGAA in the frame work of the OTKA project of my supervisors. I compared the results with already published data of previous measurements, and checked the precision of the method by examining geological standards, thus supporting the geochemical applicability of the method (Gmélíng et al. 2005; 2007b). This PGAA method has become a geoanalytical tool which is used universally, as is proved by the numerous publications that are fruits of international co-operations.

The major aim of the dissertation was to draw a conclusion about the connection between boron and other trace element variations and the subduction processes in the Carpathian Pannonian Region from the boron geochemical results of the studied calc-alkaline volcanic rocks measured with PGAA. For comparison I measured numerous alkaline basalt samples from the region (Gmélíng et al. 2007a) and their xenoliths, but also flysch, loess, metamorphic samples and Black Sea sediments (Cristache et al. 2009) as possible contaminants. Boron as a tracer of subduction fluids gives useful information about

the 700 km long, possibly subduction related, calc-alkaline volcanic arc, which is parallel to the Carpathians.

This doctoral dissertation covers the boron geochemical study of the Miocene-Quaternary calc-alkaline volcanic rocks of the Carpathian Pannonian Region, comparing the western and eastern part of the arc. In some sections of the arc the volcanic formations are parallel to, while other sections are perpendicular to the believed subduction suture zone. The boron variation and the changes in the subduction features can be followed both in space and time along the arc. Boron concentration changes were modelled for the effect of magmatic differentiation, crustal contamination and partial melting, as well as for those of fluid origin and amount, which influenced the magma source region.

## II. EXPERIMENTAL METHODS AND ANALYTICAL TECHNIQUES

To study the boron geochemistry of the examined samples, whole rock boron concentration measurements were performed with prompt gamma activation analysis (PGAA) at the Budapest Neutron Centre. The instrument is located at the end of a cold (20 K) neutron beam, extracted from the 10-MW research reactor. The flux of the neutron beam was  $1 \times 10^8 \text{ cm}^{-2} \text{ s}^{-1}$ . The maximum cross-section of the neutron beam is  $4 \text{ cm}^2$ , but it can be collimated. The neutron flux has been proved to be stable during the reactor cycles and homogeneous over the area of the beam. There is no sample preparation needed. The emitted gamma radiation was detected with a High Purity Germanium detector, surrounded by a Bismuth Germanate scintillator for suppression of Compton scattering events. The signals were processed with a multichannel analyser. The spectra were fitted with Hypermet-PC software; the element identification was performed using our prompt-gamma library and evaluated with an Excel macro.

The reliability of PGAA measurements of geological samples was checked on international geological standards, and on volcanic rocks which have been previously measured by different methods and for which data was already published. The previously published data were used to compare the PGAA with other geoanalytical methods (Gmélíng et al. 2005, 2007a,b), and to complement the results learnt from PGAA measurements. Some of the samples from the Western Carpathian volcanic arc were personally collected, while others were previously collected by other geologists and measured with XRF, ICP-AES and TIMS at the Royal Holloway University of London (Harangi et al. 2001, 2007). Petrographic studies and radiometric age measurements (by K/Ar method) of the samples from the Tokaj Mts. were performed at the Institute of Nuclear Research of the HAS (Pécskay et al. 1986). Major and trace element content of the Eastern Carpathian volcanic arc samples (Calimani, Gurghiu and Harghita Mts.) were previously examined with XRF, while their Sr- and Nd-isotope ratios were measured with TIMS at the Royal Holloway University of London (Mason et al. 1996). Most of the samples studied from the Balaton Highland volcanic field were collected by Dr. Károly Németh and Dr. Ulrike Martin, who made physical volcanology and petrographic studies (Németh & Martin 2007). Sr- and Nd-isotope data of the lower crustal xenoliths from the Balaton Highland (from Sabar and

Mindszentkállya) were measured with TIMS at the Royal Holloway University of London (Embey-Isztin et al. 2003).

The personally collected samples were measured first with PGAA. Besides the B concentration data, Cl, Nd, Sm, and Gd concentrations were measured and also those of the major element oxides. For the boron geochemical studies it is necessary to have most of the rare earth elements (REE) and some fluid mobile element concentrations. Hence I also made ICP-MS, LA-ICP MS and NAA measurements. The double-focusing inductively coupled plasma mass spectrometer (ICP MS, ELEMENT2) at the Institute of Isotopes HAS, was used with the help of Dr. Zsolt Varga (Gmélíng et al. 2007c) and Dr. Zsolt Stefánka. All measurements were made using a Scott-type spray chamber and a Meinhard concentric nebuliser. The instrument was operated in the low resolution mode ( $R=300$ ). The dissolution of the samples was carried out using a MARS5 microwave digestion system. Although, the use of the ICP-MS method is widely spread in geochemistry, the major use of the instrument in the Institute of Isotopes HAS is not for geoanalysis, and thus we measured the first geological samples in solution by the instrument. The dissolution of the samples was done following methods given in the literature (Diegor et al. 2001) and with the help of chemist colleagues.

The ICP-MS method can be extended with a Laser Ablation sampling head (LA-ICP-MS). In the frame of the cooperation of the Institute of Isotopes HAS and the Geochemical Research Institute HAS (Geoanalytical Research Group) we prepared the multielemental geochemical usage of the LA-ICP-MS method. For LA-ICP-MS measurements the sample should not be dissolved, and the studied elemental concentration is only for the volume of the sample vaporised by the laser. We explored a method of sample preparation to measure the bulk rock trace elemental concentrations with LA-ICP-MS. Dr. Zsolt Stefánka, Dr. Gábor Dobosi and Róbert Katona helped with the bulk rock LA-ICP-MS measurements. NIST-612 glass was used as a standard, while on internal standard was the Ca content measured with PGAA and XRF.

A laser beam with 70% energy and 60  $\mu\text{m}$  cross-section was used for sampling, while the transport gas was Ar. The advantage of the new method is the cheaper and easier sample preparation, but the disadvantage is the higher uncertainty. This method was used to measure the trace element content of the samples from the Mátra and the Tokaj Mts. Some of the studied samples were measured with NAA in Warsaw and two places in Budapest (Technical University Study Reactor and Budapest Research Reactor). Most of the NAA measurements were done at the Budapest Research Reactor with the assistance of Dr. András Simonits and Dr. Réka Szőke. The  $k_0$ -method was used during the measurements. Gamma spectra were collected with an HPGe-detector coupled to a loss free counter. Spectra were fitted using the Hyperlab 2002 program. The elemental concentration was calculated with the KAYZERO/SOLCOI program, while the interferences were corrected manually. The analytical methods were chosen by the infrastructural possibilities.

### III. NEW SCIENTIFIC ACHIEVEMENTS (RESULTS)

1. In the course of the research of this dissertation I made the first boron content examinations on the calc-alkaline volcanic rocks of the Carpathian Pannonian Region (Gmélíng et al. 2007c). Boron geochemical data of 150 calc-alkaline samples published in this study gives a representative picture of the boron distribution of the region. For comparison, the boron content of numerous different type and age volcanic rocks were measured.
2. The validity for geochemical use of the prompt gamma activation analysis (PGAA) was proved by the measurement of geological standards, and with the repeated measurement of many samples which had been analysed previously with different – well established geoanalytical – methods (Gmélíng et al. 2005; 2007b). Due to the good agreement between the methods, the PGAA instrument in Budapest is well accepted by the geoanalytical community. Numerous articles were published, with my co-authorship, on the subject of geochemistry using the Budapest PGAA as one of the analytical techniques: Marschall et al. 2005, 2006, 2009; Harangi et al. 2007; Pelletier et al. 2008; Németh et al. 2008; Cristache et al. 2009; Di Nicola et al. 2009, Kiss et al. 2010; Kodolányi et al. 2010; Seghedi et al. 2010a.
3. Most of the samples examined in this study were measured previously with other different analytical methods. Thus the PGAA results could be compared and complemented. I measured by NAA and LA-ICP-MS the personally collected samples and those which had no previous chemical analysis (from the Tokaj Mts. and the Mátra Mts.). Hence, I could compare the PGAA result with and also complement them, to publish absolute new results from the above mentioned samples. LA-ICP-MS measurements were done on whole rocks with a newly developed sample preparation and measurement method (powdered samples were pressed to pellets and from three laser line samplings averages were calculated). The new method simplified the sample preparation procedure, but the results are less precise compared to solution ICP-MS measurements.
4. The effect of different magmatic processes (fractional crystallisation – FC, assimilation and fractional crystallisation – AFC, and partial melting – PM) on the boron content was modelled on the most suitable rock series of the western and the eastern parts of the Carpathian volcanic arc. The boron concentration data gives useful information about the metasomatic processes influencing the source region of calc-alkaline magmas. However, the boron content alone is not enough, as it is fluid mobile and incompatible, and thus its concentration is easily changed by the different magmatic processes (FC, AFC, PM). It is useful to study the boron content relative to other similarly incompatible elements to cancel the effect of magmatic processes. A boron ratio relative to a non-mobile element can give information about the volume of the metasomatic fluid influencing the source region of the magma. The best trace element ratios to study the amount and origin of the metasomatic fluids are the following: B/La, B/Nb, or B/Sm. The effects of FC, AFC and PM processes were modelled for the above mentioned trace element ratios. FC processes have nearly no

effect on the B/Sm or on the B/Zr ratios. Assimilation of the lower crustal material is not influencing the B/Sm, B/La and the B/Pb ratios, while the assimilation of the upper crust is slightly increasing those ratios. The effect of the partial melting is stronger if the degree of the melting is very small, because then the most incompatible elements, like boron, go immediately to the melt. A larger melting ratio will lower the relative concentration of the incompatible elements, as the less incompatible elements are also going to the melt phase. Melting can be initiated by an increased amount of fluids. In this case the fluid tracer ratios (e.g. B/Nb, B/La etc.) and the ratios indicating the increasing partial melting (e.g. Zr/Nb) will show close correlation. Only for the examined volcanic areas in the Central Slovakian and the Calimani volcanic rocks are the Zr/Nb and the B/Nb ratios are both decreasing due to the metasomatic fluid initiated partial melting. Below the other examined region the melting was initiated by decompression.

5. The magma can originate from the asthenosphere, from the upper part of the mantle, or from the lower part of the lower crust. To reach a conclusion about the boron content of the various source regions, it was necessary to measure the average boron content of materials from the above mentioned possible sources. I measured the boron content of the alkaline basalts of the Carpathian Pannonian Region (Balaton Highland: 4.7-12.4  $\mu\text{g/g}$ , Gmélíng et al. 2007a; Selmechbánya: 1.7-1.8  $\mu\text{g/g}$ ; Brehy: 5.7  $\mu\text{g/g}$ ), which are representing the melted upper mantle material. I also measured xenoliths originating from the upper mantle (Balaton Highland: 0.04-0.27  $\mu\text{g/g}$ ; Persányi Mts.: 0.7-2  $\mu\text{g/g}$ ), and from the lower crust (Mindszentkál: 0.4-0.7  $\mu\text{g/g}$ ; Sabar Mt.: 0.1-0.7  $\mu\text{g/g}$ ).
6. The melt moving towards the surface can assimilate matters from the surrounding rocks and become contaminated with the lower crustal, upper mantle materials, and with the subducted sediment or slab materials and fluids originated from them. I also tried to determine the average boron content of the local upper crust by measuring different rock types referring to its composition (loess: 54.1-64.6  $\mu\text{g/g}$ ; metapelites and shist: 58.3-67.1  $\mu\text{g/g}$ ). The boron content published in the thesis gives good basic information about the average boron content of different possible assimilation source in this region, and can be used in petrogenetical models. I also measured the boron content of Cretaceous flisch sediments (112-121 and 194  $\mu\text{g/g}$ ), which are associated with the possibly subducted sediments. This showed good agreement with the boron content of the Black Sea sediment measured in an international cooperation (129  $\mu\text{g/g}$ , Cristache et al. 2009), and with the literature data of sediments from the Pacific Ocean (96-132  $\mu\text{g/g}$ , Ishikawa & Nakamura 1993). I made calculations about the boron content of fluids originating from the subducted sediments, to show that the boron concentration of fluids (5200-5600  $\mu\text{g/g}$ ) are much higher than the boron content of the sediments.
7. Results of boron geochemical studies on the rock samples of the Western Carpathians volcanic field
  - a. The boron geochemical data of the examined volcanic rocks lead to the conclusion that there is no close connection between subduction and volcanism. This hypothesis is also confirmed by Lexa & Konečný (1974) and Grad et al. (2006) who put forward the conception of an extension

related volcanism in the region. There are no systematic changes with time and space in the trace elements, especially in the boron contents, of the Western Carpathian volcanic arc, which characterises the recently active subduction zones. The only exception is the Central Slovakian volcanic field, where the boron content is decreasing as the age of the volcanic rocks is getting younger. The early volcanics (with low Nb/Y, high Zr/Nb and variable B/Nb ratios) could be formed from the melting of the lithospheric mantle, which was previously metasomatised with fluid originating from the subducted slab. With time, in the shallow lithospheric mantle, the Ba could be retained more easily than the extremely mobile B. The later volcanics (with high Nb/Y, low Zr/Nb and also low B/Nb ratios) could be formed from a less contaminated asthenospheric material.

- b. Under the Western Carpathian volcanic field, the volcanism seems to have been initiated by decompression melting, which assumption is supported by the variable trace element ratios indicating the varying degree of partial melting (e.g. Zr/Nb), and also by the almost constant trace element ratios reflecting lack of much fluid metasomatism (e.g. B/Nd). I concluded that, probably there were not enough subducted sediments, and/or the slab did not reach the depth of the melting zone, thus the amount of the metasomatising fluids was not enough to decrease the solidus of the mantle wedge material to enable it to melt. Thus the melting was not caused by the fluid metasomatism, but it was generated by the decreasing pressure. The lateral movements and shearing of the tectonic plates could cause weakened zones and decompression melting (Grad et al. 2006). The degree of the partial melting was variable under the different volcanic fields, but that variation did not correlate with the measured random variation in the fluid mobile trace element composition.
- c. Volcanic rocks of the region contain subduction components, including boron, which are indicating fluid metasomatism. Subduction fluids more probably originated from the crust, rather than from the subducted sediments, the former of which has higher Ba/Sm and K<sub>2</sub>O/Sm ratios than the B/Sm ratios. The higher Ba and the K<sub>2</sub>O are point to a crustal origin, which is not surely originating from the slab, but can be derived also from the metasomatised lower crust, or from upper crustal assimilation. The B and Ba contents are relatively higher in the examined volcanic rocks than could be formed by the melting of the upper mantle material. However, a metasomatised mantle melt could also have mixed with the lower crustal melts. The assimilation of the lower crust can increase the Ba/Sm and the K<sub>2</sub>O/Sm ratios more than B/Sm ratios. The above mentioned results support the idea of Harangi et al. (2001, 2007), who presumed that under the Western Carpathian volcanic region there was absolute mixing between the melt originating from the mantle and the melt originating from the lower crust.

## **8. Results of boron geochemical studies on the rock samples of the Eastern Carpathians volcanic field**

- a. The incompatible and fluid mobile element content of the examined rocks including B indicates the fluid metasomatic effect of the subducting slab. Under the Calimani-Gurghiu-Harghita

volcanic arc the magma source region was only partially melted. The degree of partial melting is slightly changing under the different volcanic areas, but that is not enough to cause the measured variation in the trace element content.

- b. The boron geochemical data and also the other fluid mobile and incompatible element data of the Eastern Carpathian volcanic field is changing along the arc with time and location (Pécskay et al. 1995; Mason et al. 1998), which is connected to the gradual tearing of the slab, and finally at the southern end of the arc with the changes of the physical and chemical parameters of the slab. The amount of subduction components is high, especially at the southern end of the arc. The subduction fluid has a different metasomatic effect on the different magma source regions. In the Calimani and Gurghiu Mts. medium B/Zr- and B/Sm ratios and medium Ba/Zr-, Ba/Sm-ratios exist along with low Nb content, implying a MORB-type source region (depleted mantle composition) which was metasomatised with slab derived fluids. Volcanics of the North Harghita Mts. have lower Ba/Zr- and Ba/Sm ratios than B/Zr- and B/Sm ratios, but higher Nb content, referring to a mantle source region which was contaminated with Outer Carpathian flisch sediment with high B content ( $> 100 \mu\text{g/g}$ ), or metasomatised by the fluids with even higher B content ( $\text{B} > 5000 \mu\text{g/g}$ ), originating from them.
- c. The Trotus line lies between the North- and South Harghita Mts. It is changing the geometry of the slab, and due to that, the composition of the volcanic rocks formed from it. The source region of the South Harghita Mts. was similar to an OIB-type mantle material which was metasomatised with fluids rich in Ba/Zr and Ba/Sm. The extremely high Ba content can not be explained by the FC or AFC processes, especially because the Ba content of the contaminating material is lower. The volcanic rocks of the South Harghita Mts. are of adakitic type in composition (Seghedi et al. 2004; 2010b), which leads to a conclusion that the slab could melt under this region. The slab dehydrated before reaching the melting temperature, thus most of the strongly fluid mobile B would have left the slab before melting (Defant et al. 1991; Leeman et al. 1994). The dehydrated slab contains only a small amount of B, so its melt is B depleted. The low B/Sm and the high Ba/Sm ratios of the South Harghita samples with low Zr/Nb ratios and low Y and Yb content supports the idea of Szakács et al. (1993) and Mason et al. (1996), that the slab edge next to the Trotus line could partially melt due to the upwelling asthenospheric material, which melt made a corner flow next to the steep Moesian slab, and differentiated in the lower crust (Seghedi et al. 2010b).

#### IV. CONCLUSIONS

In order to validate the results obtained using PGAA, I compared the PGAA data with the data of previous measurements, and with the examination of geological standards, and I proved the accuracy of the PGAA method. Following the first international publications, numerous co-operations were born and the Budapest PGAA became a generally accepted method in geoanalysis. I completed one of the



most important goals of the study: mapping the boron variation of the Neogene–Quaternary calc-alkaline volcanic rocks of the Carpathian Pannonian Region. Modelling the fractional crystallisation, assimilation and partial melting processes I reproduced the boron content changes of the rocks, and also the origin and enrichment of the fluid. I studied the connection between the subduction processes and the boron geochemistry of the examined rocks, supporting the previous idea of post-subduction and sin-extension volcanism and mostly extension related melting in the region. I compared petrogenetic processes and the effects of subduction related fluids on the sources using the boron geochemical data of the western and eastern part of the Carpathian volcanic arc. I made PGAA measurements on numerous alkaline basalts and xenoliths from the region, and also on some contaminants, like loess, flisch, metamorphic rocks and Black Sea sediments.

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